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MATERIALS RESEARCH FOR THE CLEAN UTILIZATION OF COAL

Quarterly Progress Report

October - December 1981

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I. SUMMARY OF PROGRESS TO DATE

Brief Summary

1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff).

The major effort this quarter has continued to be the completion of the first issue of pages of the book, "Construction Materials for Coal Conversion--Performance and Properties Data"; the first version was delivered to the DoE monitor. A talk was given at the Sixth Annual Conference on Materials for Coal Conversion and Utilization.

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

During this quarter, five fused cast refractory bricks were sectioned and examined for homogeneity. The alumina-zirconia-silica refractory was chosen for testing on the basis of its good density and its higher strength and durability during exposure. Creep rupture tests were continued on the silicon carbide samples discussed previously.

II. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

1. Materials Performance and Properties (H. M. Ondik, B. W. Christ, and A. Perloff).

Progress:

Data Center activities for the past quarter have included the regular data activities: receiving and cataloging reports, answering queries, abstracting data, etc.; as well as work on the book, "Construction Materials for Coal Conversion--Performance and Properties Data". The draft manuscript of the first issue of the book was delivered to the Department of Energy program monitor in the third week of December without the index. Further material is to be inserted in the book, and the indexes are to be completed.

A talk was presented at the Sixth Annual Conference on Materials for Coal Conversion and Utilization in October at NBS. The topic of the talk was "Information Systems for Fossil Energy Materials Applications" and presented an overview of data bases which are of use in the fossil energy field, as well as a description of the data bases of this Data Center.

Five requests for information were received from various refractories manufacturers following an announcement that refractories data for use in dry ash gasifiers generated by DoE programs is available from NBS. The announcement appeared in the November 1981 issue of The Refractories Institute Newsletter.

Plans:

The final manuscript of the book, "Construction Materials for Coal Conversion--Performance and Properties Data", will be delivered to the program monitor at DoE. The routine activities of the Data Center, with respect to receipt and handling of reports and data and the response to queries, will continue, and work will begin on an update of the book.

2. Creep and Related Properties of Refractories (N. J. Tighe, C. L. McDaniel, and S. M. Wiederhorn)

Experimental Procedures:

Slabs one inch thick were cut from the ends of the five fused cast billets which are identified as follows:

1.	alumina-zirconia-silica	Monofrax CS-3
2.	alpha-beta alumina	Monofrax M
3.	alpha alumina	Monofrax A
4.	chrome-alumina	Monofrax K-3
5.	chrome-alumina spinel	Monofrax E

The Monofrax designations are the Carborundum designations. A compilation of the material properties presented by Carborundum is given in Table 1. Values for high temperature strength are not presented for the materials.

Table 1. Properties of fused cast refractories*

	Max. Temp.	Modulus of Rupture, RT		Compressive Strength, RT		Thermal Exp.
	°C	Ksi	MPa	Ksi	MPa	cm/cm °C x 10^{-6}
CS-3	1590	9.8	68.6	20	140	7.3
M	1870	3.5	24.5	18	126	7.9
A	1930	5.8	40.9	20	140	7.9
K-3	1870	8.0	56.0	20	140	7.9
E	1930	5.7	39.9	20	140	7.4

*Carborundum

The pictures of the slabs in Figure 1 show the porosity in the central regions and the range of inhomogeneity across the slab face. From examination of the slabs, the alumina-zirconia-silica billet appears more homogeneous than the other billets. This slab was cut into ∿50 bars for flexure creep rupture tests. Bars 5 x 10 x 50 mm were cut with the two orientations shown in Figure 2. Machined specimens were heated several hours at 800 °C to remove the grinding oils. The manufacturers

analysis in wt. percent is: 50.1 Al_2O_3, 34.2 ZrO_2, 14.2 SiO_2, 1.1 Na_2O, 0.12 Fe_2O_3, 0.05 TiO_2. Phases identified from powder X-ray diffraction patterns made of the as-received material were alpha alumina, monoclinic zirconia and an amorphous silica phase. Samples that were heated for 24 hours at 1200 °C, 1300 °C, and 1400 °C had the same powder diffraction patterns.

The creep tests on the silicon carbide samples which were being run for 1000 hour periods were terminated after several hundred hours because of water cooling problems. A closed cycle cooler was purchased and is being installed.

Plans:

The flexure strength will be obtained at room temperature and at 1200 °C for the alumina-zirconia-silica specimens. From these results, the static loads for the creep experiments will be selected. Annealing conditions required for the precipitation of the tetragonal phase of ZrO_2 will be identified. The phase composition and the microstructural changes will be identified and monitored using powder X-ray diffraction and analytical electron microscopy.

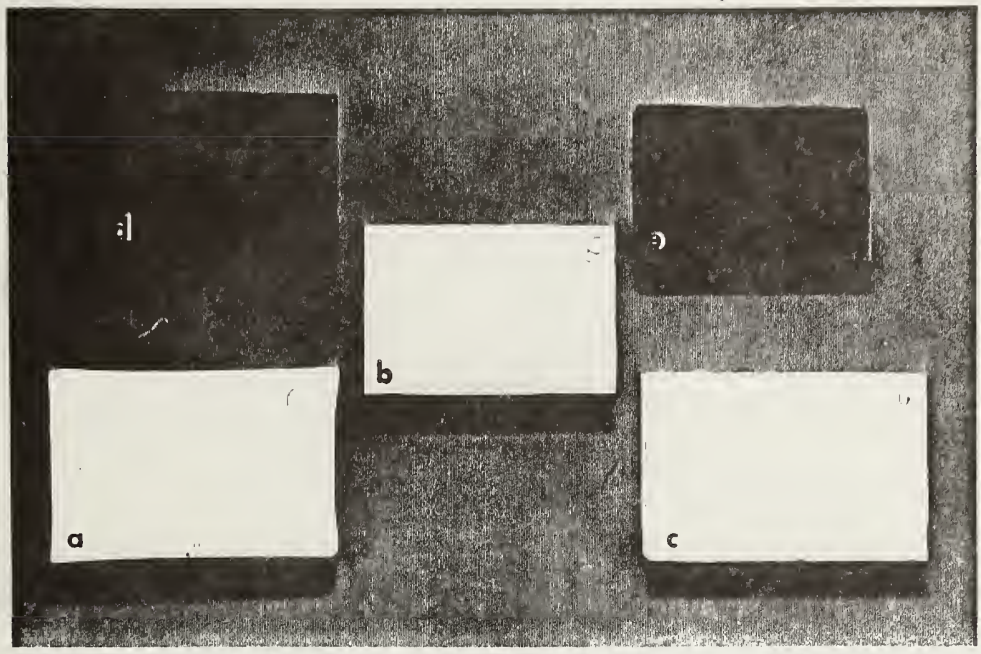

Figure 1. Cross-section of slabs from fused-cast billets a) alumina-zirconia-silica, b) α-β alumina, c) α alumina, d) chrome alumina, e) chrome-alumina spinel.

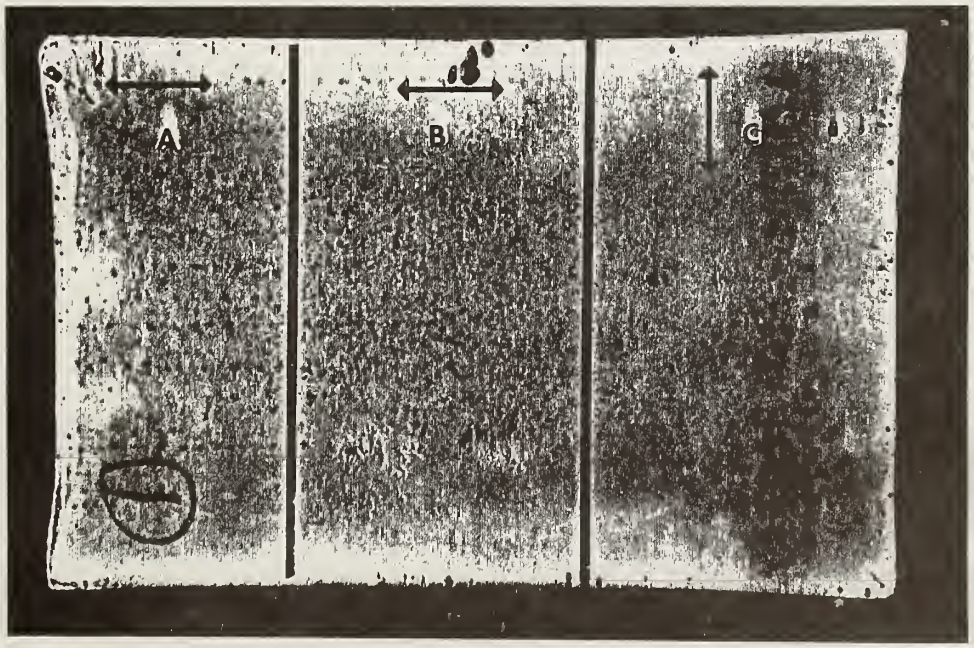

Figure 2. Section of billet of alumina-zirconia-silica showing orientation of test bar specimens.